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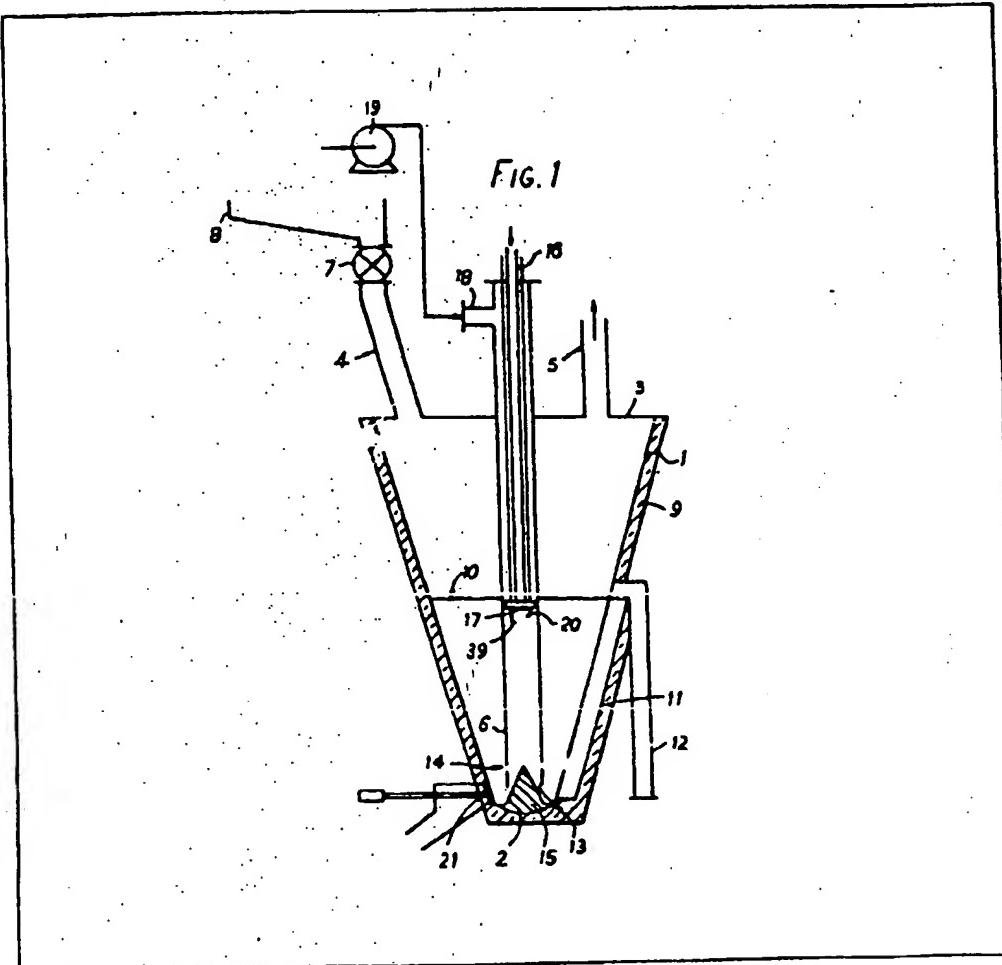
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(54) Calcination method and apparatus

(57) Minerals such as gypsum in powdered condition are calcined with increased thermal efficiency in a vessel having a lower region of small cross section, by means of hot gas delivered by a downwardly extending tube directly into the lower region of the vessel.

The preferred vessel is an inverted cone (1) and has a lid (3) which is fitted with a mineral inlet (4) and exhaust outlet (5), and through which the hot gas tube (6) passes. The hot gas may be produced in the tube by a burner (17). Discharge from the vessel is preferably through an overflow weir (11) and an internal protuberance (15) may be used to assist in distributing the gas flow across the bottom of the vessel to avoid accumulation of calcined material.

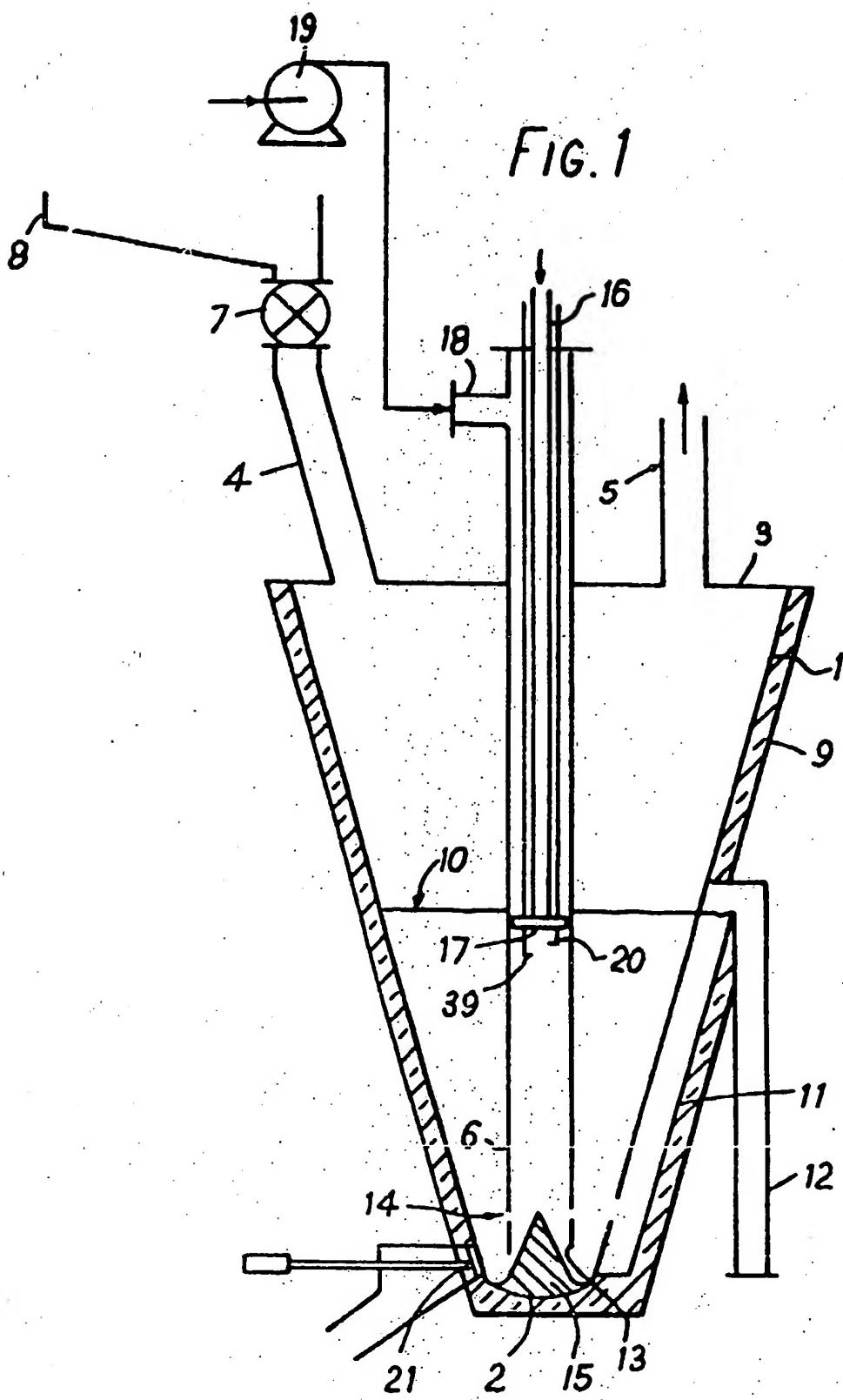


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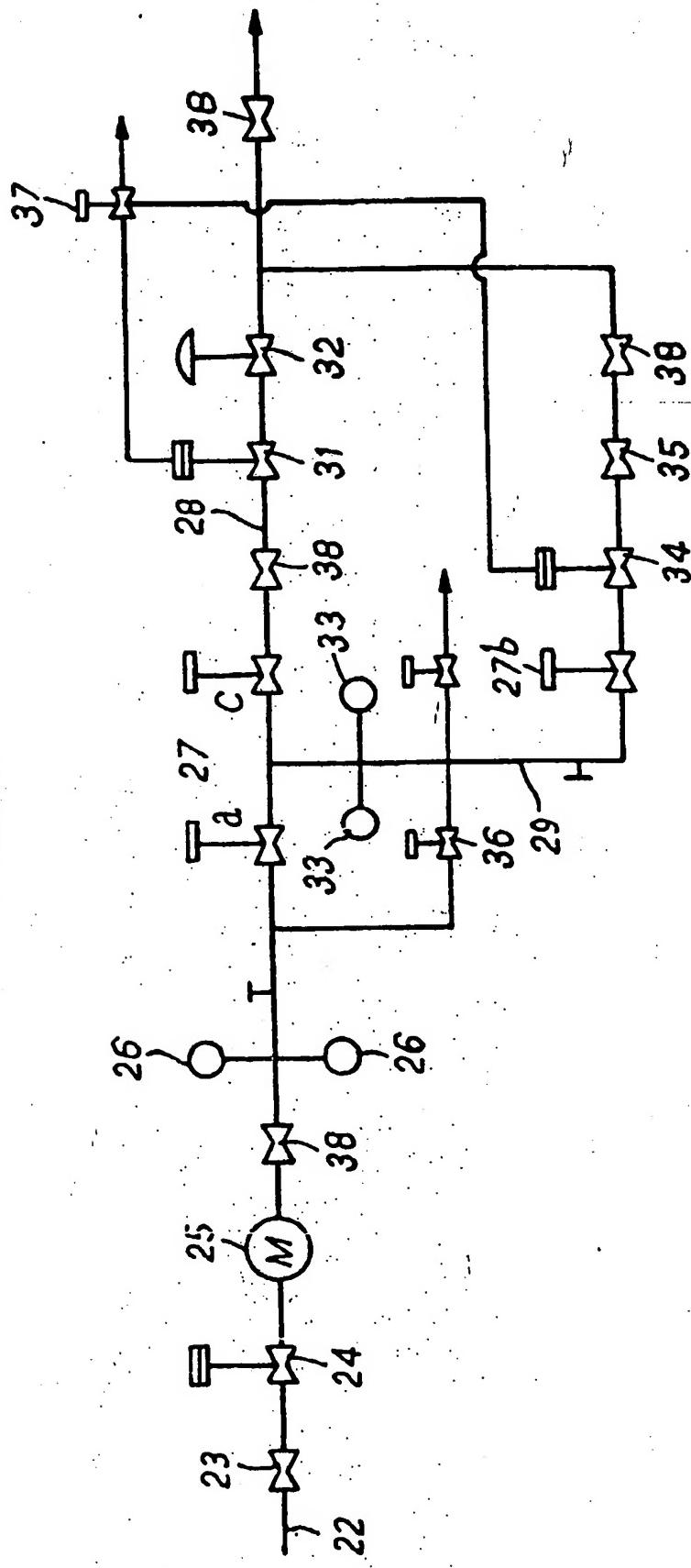
FIG. 1



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FIG. 2



**SPECIFICATION****Calcination method and apparatus**

5 This invention relates to a method and apparatus for heat-treating particulate material, especially minerals, and more particularly for calcining gypsum (hydrated calcium sulphate).

One of the main methods by which gypsum 10 calcination is carried out industrially is in a so-called "kettle". Calcination kettles have conventionally been operated on a batch basis, but more recently they have been operated by a continuous technique, as described in British Patent No. 1 018 464.

15 However, the production rate in both batch and continuous kettle calcination is at present limited by the maximum permissible heat transfer through the kettle walls, and especially through the bottom. This heat transfer is limited because there is a maximum 20 permitted temperature for the metal of the kettle walls and bottom, usually steel. Above this maximum there is a danger of frequent bottom burn-outs.

The invention described in our British Patent No. 1 25 488 665 aimed to increase the heat input to calcination kettles in both batch and continuous operation, thereby increasing the production capacity of the kettle, without adversely affecting the product quality or significantly increasing the kettle bottom 30 temperature. According to that invention, calcium sulphate dihydrate is heated in a calcination vessel or kettle by heat applied indirectly through the external walls and bottom of the vessel and, additionally, by the direct introduction of hot gas into the 35 vessel through a tube extending downwardly from the top of the vessel and provided with at least one opening in its lower region which is immersed in the mass of the calcining material. While the application of the method disclosed in that patent has given 40 greatly increased throughput and substantial increases in the thermal efficiency of kettles, the kettle itself is still subject to the maintenance problems usual with such vessels, and may occasionally require extensive repairs, for example to the refractory brickwork.

We have now developed a new method and apparatus for heat-treatment of particulate, i.e. powdered or granular material and especially suitable for calcination, in which all the necessary heat can be 50 supplied directly into the material being calcined, thereby making it possible to dispense with much of the refractory brickwork associated with conventional kettles and to thermally lag the system, so increasing the thermal efficiency and reducing capital 55 and maintenance costs.

According to the present invention there is provided a method of heat-treating particulate material which comprises introducing the material into a vessel, restricting material in the bottom region of 60 the vessel to the vicinity of the hot gas outlet, introducing hot gas downwardly through the vessel into direct contact with the material in the region of the bottom, whereby the material at the bottom is simultaneously heated and circulated and the resulting agitation and heating extends from the bottom 65

region through substantially all the material in the vessel, and withdrawing heat-treated material from the vessel.

Further, in accordance with the present invention, 70 there is provided apparatus for heat-treating particulate material comprising a vessel having an imperforate bottom which is contacted by the contents of the vessel when in use, an inlet for the material to be heat-treated, an outlet for heat-treated material, and 75 at least one downwardly extending heating tube adapted for the passage of hot gases and opening into the interior of the vessel adjacent to the bottom, the bottom of the vessel being shaped to restrict material at the bottom to the proximity of the tube opening, whereby in use hot gas issuing from the lower portion of the heating tube will simultaneously heat and circulate the material at the bottom and thence agitate and heat substantially all the contents of the vessel. It is envisaged that in most cases the 80 hot gases will provide all the heat required for treatment in the vessel, although some degree of wall heating may be provided without departing from the principles of the invention.

The vessel may have any convenient shape, 85 provided that its bottom is restricted in area and shaped to ensure that solids on the bottom are caused to circulate by upward flow, thereby bringing about agitation of the entire mass of material in the vessel and efficient transfer of heat from the gases. 90 In preferred forms of vessel, the bottom has a sufficiently small cross-sectional area, in relation to the size and position of the hot gas tube, to ensure that the gas issuing from the tube sweeps the particulate material across the entire bottom of the 95 vessel, preventing the accumulation of heat-treated material, and ultimately mixing substantially the whole contents of the vessel.

Although asymmetrical vessels can be used, subject to the requirement for good mixing, it is 100 preferred that the vessel be substantially symmetrical about a vertical axis, and the heating is preferably arranged to lie substantially along such vertical axis. A particularly preferred form of vessel at present is an inverted conical or frusto-conical vessel, more 105 especially one in which the walls are upwardly divergent at the level of the surface of the material in the vessel in operation.

The shape of the bottom region of the vessel is 110 less critical, in that a cylindrical bottom region may suffice if it is of small enough diameter, in relation to the gas tube, to ensure clearance of solids from the bottom in operation. A wholly cylindrical vessel of such diameter is possible, especially if the angle between the side and the base is faired to improve 115 smoothness of flow, but in order to obtain the necessary residence time to ensure adequate heat-treatment, especially in the case of calcination, it may have to be of uneconomic height. Moreover, an upper region of enlarged diameter or cross-sectional 120 area assists in reducing the amount of fine material lost in the exhaust gases as dust, which then requires separation from the exhaust. For this reason a vessel with a substantial conical or frusto-conical portion is preferred, although this may be 125 combined with a narrow cylindrical bottom region.

surrounding the bottom of the hot gas tube, or a wide cylindrical upper portion to assist in dust separation.

British Patent 1 240 655 describes a method and apparatus for calcining a granular calcareous material (that is, lime), in which the hot gases from a conventional fluidized bed calciner are circulated to a pre-heater having the form of a cylindrical vessel with an inwardly tapered lower part, the gases being introduced through a tube extending downwardly through the vessel to an open end near the base. It must be noted, however, that the invention differs fundamentally from this proposal. Whereas it is important for the purposes of the present invention

that heat-treated material should not be allowed to accumulate at the bottom of the vessel, this is of no significance in the process of the British Patent, where the preheater operates at a fraction of the reaction temperature, and the extent and uniformity of the heating effect are in no way critical. The bottom of the preheater is not designed to avoid stagnant zones. Indeed, the likelihood of the accumulation of dust is envisaged, and the provision of a conventional perforated base for the introduction of fluidizing gas is recommended for the purpose of dispersing it.

It may also be mentioned that the present invention is to be distinguished from so-called 'spouted bed' reactors, in which the gas is introduced in an upward direction through an opening at the apex of the inverted conical lower portion of the reactor vessel. It is believed that the pattern of flow of material in the apparatus of the present invention is largely the reverse of that in a spouted bed reactor.

The bottom of the vessel may be specially shaped to assist in distributing the flow of hot gases across the bottom surface or may be provided with one or more internal protuberances or inserts to serve this purpose. For example, a conical insert may be seated on the vessel bottom, having its apex directed upwardly towards the lower opening in the heating tube. The bottom of the vessel, or more especially an internal protuberance as described, may be fitted with baffles or swirl vanes to enhance the distribution and effectiveness of the gas flow. It is also advantageous to avoid discontinuities in the profile of the surface, for example by providing a curved profile at the junction between the bottom and a conical insert.

The heating tube is preferably open at its lower end. In addition, or even alternatively, it may have in its lower part a plurality of gas distribution holes in the side wall, themselves preferably symmetrically disposed. It may also have side openings at a higher level, to give additional heating or agitation at such level, provided there is sufficient flow of gas at the bottom of the vessel to avoid accumulations of heat-treated material.

The heating tube may be connected to a source of hot gases at an appropriate temperature, which may be derived from the combustion of a fuel, for example, gas, fuel oil or coal, or may be hot exhaust gases from another process, provided it does not interfere with a desired calcination reaction or product.

Alternatively, and in many cases preferably, the heating tube may be connected in its upper portion to a supply of fuel, which may itself be gaseous, and also to a source of oxygen and/or air, in which case the tube will include a fuel burner to produce hot gaseous combustion products. The burner may be provided with a combustion-initiating device, which may for example be electric. By way of example, the fuel may be town gas or natural gas, and the

combustion-initiating device may include a spark device. The fuel burner is preferably positioned in the lower half of the tube so that in use the combustion of the fuel occurs at, or below, the level of the material in the vessel.

The vessel is preferably lagged externally against heat losses, in order to enhance the thermal efficiency of the system. It will also, desirably, be connected in its upper region to a dust collector. When the invention has been used for calcination, it has been found that the dust which is collected in use, particularly in the case of gypsum, is predominantly composed of calcined material and is itself a useful product.

If the vessel is to operate on a continuous basis, it is preferably provided with a valved inlet for the material, such as calcium sulphate dihydrate, and a valved outlet or overflow system for heat-treated material. Any suitable technique may be used for controlled feeding of material into the vessel or for discharging material out of the vessel. Achievement of a fully reacted and uniform product requires, as well as an adequate residence time, assurance that an unacceptable proportion of unreacted or insufficiently reacted material does not reach the outlet. If

the reaction is fast and the outlet sufficiently remote from the inlet (for example, in a vessel of large diameter at the level of the top of the material) this may suffice. In most cases however, it is preferred to fit means for extending the path between the inlet and the outlet. These means may comprise one or more baffles arranged round the outlet or the inlet or between them, or tubes or conduits leading to or from the outlet or inlet and opening at a lower level in the vessel.

An overflow system may comprise a rising discharge conduit leading from a lower region of the vessel to a weir over which discharged material flows. A rising conduit may function effectively because of the fluidization of the material in the

vessel during use. This fluidization may be brought about directly by the action of gases from the heating tube, but where gases or vapours are given off by the material being treated, these may assist or be largely responsible for fluidization of the material in the vessel. For example, gypsum loses water to give hemihydrate or anhydrous calcium sulphate, and the water vapour evolved causes "boiling" of the mineral.

The fluidization of the treated material in the apparatus of this invention, whether due primarily to the incoming gas or to self-fluidization by evolved vapours, is responsible for rapid and efficient mixing of the contents and heat transfer, and also facilitates even discharge of the product in continuous operation. Because of this action it is not necessary for the

vessel to be equipped with mechanical agitators or stirrers. However, the provision of stirrers for additional agitation does not avoid or vitiate the principles of this invention.

- 5 Although reference is frequently made herein to continuous operation, for which the apparatus is particularly intended, it should be understood that batch operation of the invention is equally possible. For this purpose the vessel is first charged with 10 material, if necessary with an initial gas flow, whereafter gases at the desired temperature are injected until the process is complete. The contents of the vessel are then discharged, for example through a bottom discharge gate. The latter is in any 15 case a desirable fitting, and can be used in cleaning the vessel and for dumping of its contents in an emergency. For cleaning and maintenance purposes, the vessel may also be provided with inspection or access hatches.
- 20 The temperature of the gases within the heating tube and issuing therefrom can be controlled in a number of ways, for example by the quantity of excess air used in combustion. Alternatively, an auxiliary air inlet may be provided in the tube 25 between the burner and the lower region of the tube whereby additional air can be introduced to control the temperature of the mixture of air and combustion products in the tube.

The production of hemihydrate plasters and 30 anhydrous plasters or mixtures thereof can be carried out by this method mainly by controlling the effective calcination temperature. For example, if the temperature of the mass of the calcium sulphate being treated is maintained at about 140° to 170°C, 35 the principal calcined product from calcium sulphate dihydrate is the hemihydrate, whereas at a much higher temperature, notably around 350°C or above, the principal product is anhydrous calcium sulphate.

Where an anhydrous product is required, the 40 calcination reaction may be carried out in a single stage, by adjusting the throughput in relation to the gas temperature and flow rate to maintain a temperature in the range mentioned. However, it is also possible to conduct the reaction in two stages, by 45 operating a first apparatus according to the invention in the lower temperature range to obtain a hemihydrate product, and a second apparatus at the higher temperature. With this arrangement, water vapour is evolved at each stage to assist in fluidization and mixing. The exhaust gases from the second apparatus may be used to heat the first, and all or part of the hemihydrate product from the first apparatus may be transferred to the second. The 50 hemihydrate and anhydrous products may be utilized separately, or may be mixed in desired proportions for use as a mixed plaster.

The invention is especially suited to the calcination of natural gypsum or chemical gypsum of whatever source, e.g. synthetic gypsum from the manufacture 60 of phosphoric acid or from the neutralisation of flue-stack gases or from the hydration of natural anhydrite.

The invention will now be described, by way of example, with reference to the accompanying drawings in which:-

Figure 1 is a schematic diagram of a conical calcination vessel according to the invention, suitable for calcining gypsum, and

- 70 Figure 2 is a schematic diagram of one example of a control system for operating the heating tube of the conical vessel of Figure 1.

Referring firstly to Figure 1, a vessel 1 of inverted conical form has a rounded bottom 2 of restricted area and a lid 3 fitted with a feed pipe 4 for the 75 material to be calcined, such as powdered gypsum, and an exhaust gas outlet pipe 5 connected to a dust collector (not shown). A heating tube 6 which is described in more detail below, also passes through the lid 3 into the interior of the vessel. The gypsum feed pipe 4 is provided with a metering valve in the 80 form of a rotary feeder 7, which is connected to a gypsum bunker 8. The vessel is suitably lagged as indicated at 9.

The normal level of powdered material in the 85 vessel when it is operating is indicated at 10. An outlet for calcined material is provided in the form of an external overflow weir 11 connected to a lead-off pipe 12. A valved bottom discharge gate 21 is also provided.

90 The heating tube 6 extends downwardly substantially along the central vertical axis of the conical vessel 1. It is open at its lower end 13 and terminates adjacent to the bottom 2, of the vessel. The tube is also provided in the lower part of its wall with 95 symmetrically disposed holes 14 for further facilitating the distribution of hot gases into the material being calcined. The distribution of gases emerging from the tube 6 can be further enhanced by the provision of an upright cone 15 of heat-resistant material, which is seated on the bottom 2 of the vessel directly beneath the tube opening 13.

The dimensions of the vessel and the conical angle may be varied, in relation to the temperature and rate of flow of the hot gases through the tube 6 and 105 the intended throughput of mineral, to achieve the desired residence time and temperature of material in the vessel and to ensure adequate mixing and efficient heat transfer.

Fuel gas, for example natural gas, is supplied 110 through a pipe 16 to a gas burner 17 of a nozzle-mix type located within the tube 6 at approximately the level 10 of the material in the vessel. Air is supplied separately to this burner through an air pipe 18 from a fan 19. The fuel/air mixture leaving the nozzle mix 115 burner 17 is ignited by a spark probe 20 and the hot, gaseous products of combustion pass downwardly through the tube 6 and leave through its open end 13 and the holes 14. An auxiliary air supply to the heating tube is not generally used with this system.

120 Referring now principally to Figure 2, the fuel gas is supplied along a line 22 through a main isolating valve 23, a mains governor 24, which reduces the line pressure to a suitable figure (in this particular unit 3 psig), and then through a meter 25. The 125 pressure is then monitored by two pressure switches 26, which are set to determine the maximum and minimum pressure for safe operation. The flow of gas to the burner is controlled from a safety aspect by three shut-off valves one of which (27a) follows 130 the pressure switches 26, one (27b) is in a main line

28, and one (27c) is in a pilot line 29. The main line also includes a further governor 31 and a throughput control valve 32, and the pilot line contains a pair of pressure switches 33, its own governor 34 and a limiting valve 35. A bleed valve 36 additionally connects the main supply line to the pilot line 29, and a vent valve 37 is connected to the governors 31 and 34. Manual valves 38 are provided, which are usually left open but can be shut to isolate portions of the system. Both the main line 28 and the pilot line 29 deliver fuel gas to the pipe 16 of the heating tube 6.

Before the light-up sequence starts the shut-off valves 27 are checked to confirm that they are closed and not leaking. When the light-up sequence 15 is started, by turning on the burner switch, the vent valve 37 closes and a timer unit within the burner control panel (not shown) monitors the sequence using the two pressure switches 33 and 26 to ensure that the space between the three shut-off valves 27 20 does not become pressurised through leakage of gas or air, and after thirty seconds to ensure that when the space is pressurised by opening of bleed valve 36 that the pressure is held for a further thirty seconds.

25 If both these checks are satisfactory, then a visual indication that the check is complete will appear and the control cycle moves to the next stage. If either check has proved unsatisfactory then a warning indicator will appear. After the check indicator 30 appears there is a pause for five minutes, during which time the air will purge the burner and calciner system. When that time has elapsed, a burner programming unit in the control panel will start the ignition sequence. A spark ignites the pilot gas after 35 opening of the first shutoff valve 27a and a pilot valve 27b and the presence of flames is detected by a flame detection probe 39 approximately three inches from the burner 17. If a flame is detected and is stable, then the programming unit will allow the 40 second main shutoff valve 27c to open, bringing on the main flame. At this point the vent valve 37 closes, allowing the governor 31 to function. The flow rate of gas is controlled by the setting of the throughput valve 32.

45 The vertical position of the burner 17 in the tube may be adjustable to permit formation of the flame at any desired level in relation to the vessel and its contents.

Some advantages of the calcination equipment 50 according to this invention are as follows:

- (1) The capital cost of the system for a given throughput is lower than for a conventional kettle on account of the relative compactness and simplicity of the vessel.
- 55 (2) The unit need not use a stirrer, and hence there can be an overall saving in electrical energy.
- (3) The thermal efficiency, for example at 85-90%, is even higher than with the invention described in British Patent No. 1 488 665.
- 60 (4) The maintenance costs will be lower.
- (5) The start-up time is much shorter than with a conventional kettle, being for example approximately 10 minutes.

As to the product, the physical characteristics in 65 the case of calcined gypsum are similar to those of

the calcined material produced according to British Patent No. 1 488 665. More particularly, when the temperature of the hot gases is adjusted so that the temperature in the interior of the calcium sulphate is about 140° to 170°C the product is substantially all hemihydrate, with little soluble anhydrite and almost nil gypsum as measured by differential thermal analysis.

## 75 CLAIMS

1. A method of heat-treating particulate material which comprises introducing the material into a vessel, restricting material in the bottom region of the vessel to the vicinity of the hot gas outlet, introducing hot gas downwardly through the vessel into direct contact with the material in the region of the bottom, whereby the material at the bottom is simultaneously heated and circulated and the resulting agitation and heating extends from the bottom region through substantially all the material in the vessel, and withdrawing heat-treated material from the vessel.
2. A method as claimed in claim 1 wherein the gas emerging from the outlet sweeps the bottom of the vessel and prevents the accumulation of heat-treated material.
3. A method as claimed in claim 2 wherein the gas emerging from the outlet is given a helical swirling motion about the vertical axis of the vessel.
4. A method as claimed in claim 1, 2 or 3 wherein the material at the bottom of the vessel is restricted to a smaller cross-sectional area than material higher up the vessel.
- 100 5. A method as claimed in claim 4 wherein the material is contained in an inverted conical or frusto-conical vessel or portion of the vessel.
6. A method as claimed in any of claims 1 to 5, wherein the material is continuously introduced into and withdrawn from the vessel.
- 105 7. A method as claimed in claim 6 wherein the point of introduction of material into the vessel is remote from the point of withdrawal of the material from the vessel.
- 110 8. A method as claimed in claim 7 wherein the calcined material is withdrawn from the vessel from the lower region of the vessel by means of a weir.
9. A method as claimed in any preceding claim 115 wherein powdered hydrated calcium sulphate is calcined to a lower degree of hydration.
10. Apparatus for heat-treating particulate material comprising a vessel having an imperforate bottom which is contacted by the contents of the vessel when in use, an inlet for the material to be 120 heat-treated, an outlet for heat-treated material, and at least one downwardly extending heating tube adapted for the passage of hot gas and opening into the interior of the vessel adjacent to the bottom, the bottom of the vessel being shaped to restrict material at the bottom to the proximity of the tube opening, whereby in use hot gas issuing from the lower portion of the heating tube will simultaneously heat and circulate the material at the bottom and thence agitate and heat substantially all the contents 125 of the vessel.

11. Apparatus as claimed in claim 10 wherein the bottom of the vessel is shaped to provide a smooth profile for passage of hot gasses from the lower part of the tube whereby the bottom is continually swept clear of material.
12. Apparatus as claimed in claim 10 wherein the bottom of the vessel is provided with at least one internal protuberance disposed beneath the open bottom end of the heating tube.
13. - Apparatus as claimed in claim 12 wherein the protuberance is provided with helical ribs or swirl vanes.
14. Apparatus as claimed in claim 10, 11 or 12 wherein the side walls of the vessel are at least in part inclined, whereby the bottom is of smaller cross-sectional area than the region of the vessel at the level of material determined by the outlet.
15. - Apparatus as claimed in claim 10 or 14 wherein the vessel is at least in part of inverted conical form, and the heating tube is arranged to lie substantially along its vertical axis.
16. Apparatus as claimed in any of claims 10 to 15 wherein the vessel is thermally lagged to minimize conduction of heat through its walls.
17. Apparatus as claimed in any of claims 10 to 16 wherein the vessel is provided with structure defining an extended or indirect path between the material inlet and outlet.
18. Apparatus as claimed in any of claims 10 to 17 wherein the heating tube is open at its lower end and has a plurality of gas distribution holes in the lower part of its side wall.
19. Apparatus as claimed in any of claims 10 to 18 wherein the heating tube is connectable at an upper portion to a supply of fuel and to a source of oxygen-containing gas and includes a fuel burner.
20. An apparatus as claimed in claim 19 wherein the burner is vertically adjustable in the heating tube.
21. A method of calcining particulate material substantially as hereinbefore described with reference to the accompanying drawings.
22. An apparatus for heat-treating particulate material substantially as hereinbefore described with reference to the accompanying drawings.
23. Calcined calcium sulphate obtained by a method as claimed in claim 9 or in an apparatus according to claim 10.